

CLAIMS

- 5 1 - A process for controlled radical homopolymerisation, in an aqueous solution, of acrylic acid and its salts, or of copolymerisation, in aqueous solution, of acrylic acid with one or more hydrosoluble monomers, characterised in that it is in batch or semi-batch mode, and in that it comprises two stages, the first of which is synthesis "in situ" of the hydrosoluble transfer agent used in the second stage of
10 polymerisation.
- 2 - A process according to the claim 1 characterised in that the reactive medium of the first stage of synthesis of the transfer agent and of the second stage of polymerisation is identical and solely water.
- 15 3 - A process according to anyone of claims 1 or 2 characterised in that it is a process of controlled radical homopolymerisation, in an aqueous solution, of acrylic acid and that it is undertaken in batch mode.
- 20 4 - A process according to one of the claims 1 to 3 characterised in that the hydrosoluble transfer agent is an α -substitute β -carboxylate xanthate salt, preferably an α -substitute β -carboxylate sodium xanthate and very preferably an α -methyl β -carboxylate sodium xanthate.
- 25 5 - A process according to one of the claims 1 to 4, characterised in that in the second stage of polymerisation the limits of quantity of transfer agent are determined such that the molar ratio of transfer agent to monomer is between 0.001% and 20%, and the mass ratio of transfer agent to monomer is between 0.01% and 60%.
- 30 6 - A process according to one of the claims 1 to 5, characterised in that it consists in putting in contact in the first stage:
- a potassium xanthate,

- 2-bromopropionic acid sodium salt,
- water,

5 and then in adding in a second stage acrylic acid and at least one hydrosoluble initiator of free radicals.

7 - A process according to one of the claims 1 to 6, characterised in that the first stage is undertaken with equimolar quantities of potassium xanthate and the sodium salt of 2-bromopropionic acid.

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8- A process according to one of the claims 1 to 2 and 4 to 7, characterised in that the hydrosoluble copolymerised monomers are chosen from methacrylic acid, itaconic acid, maleic acid, 2-acrylamido-2-methyl-1-propane sulphonic acid in acid form or partially neutralised, 2-methacrylamido-2-methyl-1-propane sulphonic acid in acid form or partially neutralised, 3-methacrylamido-2-hydroxy-1-propane sulphonic acid in acid form or partially neutralised, allylsulphonic acid, methallylsulphonic acid, allyloxybenzene sulphonic acid, methallyloxybenzene sulphonic acid, 2-hydroxy-3-(2-propenyloxy)propane sulphonic acid, 2-methyl-2-propene-1-sulphonic acid, ethylene sulphonic acid, propene sulphonic acid, 2-methyl sulphonic acid, styrene sulphonic acid, as well as all their salts, vinyl sulphonic acid, sodium methallylsulfonate, sulfopropyl acrylate or methacrylate, sulfomethylacrylamide, sulfomethylmethacrylamide or from among acrylamide, methylacrylamide, n-methylolacrylamide, n-acryloylmorpholine, ethylene glycol methacrylate, ethylene glycol acrylate, propylene glycol methacrylate, propylene glycol acrylate, propene phosphonic acid, ethylene or propylene glycol acrylate or methacrylate phosphate, or from among vinylpyrrolidone, methacrylamido propyl trimethyl ammonium chloride or sulphate, trimethyl ammonium ethyl chloride or sulphate methacrylate, as well as their acrylate or acrylamide counterparts, whether quaternised or not, and/or ammonium dimethyldiallylchloride, as well as mixtures thereof.

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9 - A hydrosoluble transfer agent used in the process according to one of the claims 1 to 8 characterised in that it is obtained in the polymerisation reactive medium, namely water.

10 - A hydrosoluble transfer agent according to claim 9 characterised in that it is chosen from among the α -substitute β -carboxylate xanthate salts, preferably from among the α -substitute β -carboxylate sodium xanthates, and very preferably in that
 5 it is an α -methyl β -carboxylate sodium xanthate.

11 - A polymer of acrylic acid and of its salts, characterised in that it is obtained by the process according to anyone of the claims 1 to 8, and in that it has an average molecular mass by weight (M_w) of between 1000 g/mole and 60,000 g/mole
 10 measured by the GPC method, using as a standard 5 standards of sodium polyacrylate, and in that it has a polymolecularity index of less than or equal to 2 for a conversion rate relative to acrylic acid higher than 90% determined according to an HPLC method.

12 - A polymer of acrylic acid and of its salts according to claim 11, characterised in that it has an average molecular mass by weight (M_w) of between 4500 g/mole and 8000 g/mole measured by the GPC method, using as a standard 5 standards of sodium polyacrylate, and in that it has a polymolecularity index of less than or equal to 2 for a conversion rate relative to acrylic acid higher than 90% determined
 15 according to an HPLC method.
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13 - A polymer of acrylic acid and of its salts according to one of the claims 11 to 12 characterised in that it is a homopolymer of acrylic acid.

14- A polymer of acrylic acid and of its salts according to one of the claims 11 to 12, characterised in that the hydrosoluble copolymerised monomers are chosen from methacrylic acid, itaconic acid, maleic acid, 2-acrylamido-2-methyl-1-propane sulphonic acid in acid form or partially neutralised, 2-methacrylamido-2-methyl-1-propane sulphonic acid in acid form or partially neutralised, 3-methacrylamido-2-
 25 hydroxy-1-propane sulphonic acid in acid form or partially neutralised, allylsulphonic acid, methallylsulphonic acid, allyloxybenzene sulphonic acid, methallyloxybenzene sulphonic acid, 2-hydroxy-3-(2-propenyloxy)propane sulphonic acid, 2-methyl-2-propene-1-sulphonic acid, ethylene sulphonic acid, propene sulphonic acid, 2-methyl sulphonic acid, styrene sulphonic acid, as well as
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all their salts, vinyl sulphonic acid, sodium methallylsulfonate, sulfopropyl acrylate or methacrylate, sulfomethylacrylamide, sulfomethylmethacrylamide or from among acrylamide, methylacrylamide, n-methylolacrylamide, n-acryloylmorpholine, ethylene glycol methacrylate, ethylene glycol acrylate, propylene glycol methacrylate, propylene glycol acrylate, propene phosphonic acid, ethylene or propylene glycol acrylate or methacrylate phosphate, or from among vinylpyrrolidone, methacrylamido propyl trimethyl ammonium chloride or sulphate, trimethyl ammonium ethyl chloride or sulphate methacrylate, as well as their acrylate or acrylamide counterparts, whether quaternised or not, and/or ammonium dimethyldiallylchloride, and mixtures thereof.

15 - A polymer of acrylic acid and its salts, according to any one of the claims 11 to 14, characterised in that it is in its acid form, or in that it is totally or partially neutralised by one or more monovalent, divalent, trivalent neutralisation agents, or neutralisation agents of higher valency, or mixtures thereof.

16 - A polymer of acrylic acid and its salts according to anyone of claims 11 to 15, partially or totally neutralised, characterised in that the monovalent agents are chosen from the group constituted by compounds containing alkaline cations, particularly sodium and potassium, or lithium, ammonium, or the primary or secondary aliphatic and/or cyclic amines such as ethanolamines, mono and diethylamine or cyclohexylamine, and characterised in that the divalent, or trivalent neutralisation agents, or agents of higher valency, are chosen from the group constituted by compounds containing divalent cations belonging to the alkaline earths, particularly magnesium and calcium, or zinc, and by trivalent cations, including in particular aluminium, or by certain compounds containing cations of higher valency.

17 - A polymer of acrylic acid and of its salts according to claim 16 characterised in that it is a homopolymer of acrylic acid which is totally neutralised by soda, or totally neutralised by a soda-lime mixture in a 50/50 molar ratio, or partially neutralised by a soda-lime mixture in a 50/40 molar ratio.

18- Use of the polymer according to anyone of the claims 11 to 17 in the paper field and in particular in paper coating and mass-filling of paper, oil, paint, water treatment, detergency, ceramics, cements or hydraulic binders, public works, inks and varnishes, sizing of textiles or finishing of leather, and more specifically as a
5 dispersant and/or grinding aid agent of mineral materials such as natural calcium carbonate, precipitated calcium carbonate, kaolin, titanium dioxide or clays.

19 - An aqueous suspension of mineral fillers characterised in that it contains the polymer according to anyone of the claims 11 to 17 and more specifically in that it
10 contains 0.05% to 5% by dry weight of the said polymer relative to the total dry weight of the mineral fillers.

20 - An aqueous suspension of mineral fillers according to claim 19 characterised in that the mineral filler is chosen from among natural calcium carbonate such as
15 calcite, chalk or marble, synthetic calcium carbonate, also called precipitated calcium carbonate, dolomites, magnesium hydroxide, kaolin, talc, gypsum, titanium oxide, or aluminium hydroxide.

21 - A manufactured and/or coated paper characterised in that it contains the
20 aqueous suspension of mineral fillers according to anyone of claims 19 or 20.

22 - A paint formulation characterised in that it contains the aqueous suspension of mineral fillers according to anyone of claims 19 or 20.